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DOCUMENT-IDENTIFIER: US 4150101 A

TITLE: Process for the production of silica with an aerogel type structure

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INVENTOR-INFORMATION:

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APPL-NO: 05/ 558445

DATE FILED: March 14, 1975

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COUNTRY APPL-NO APPL-DATE

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	U	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C	P	
1	<input type="checkbox"/>	US 20040132846 A1	20040708	21	Methods and compositions for preparing silica aerogels	521/99	521/113; 521/117;		Leventis, Nicholas et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
2	<input type="checkbox"/>	US 6121336 A	20000919	10	Surfactants for incorporating silica aerogel	521/112	521/99; 524/588;		Okoroafor; Michael O. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
3	<input type="checkbox"/>	US 5911658 A	19990615	8	Method for producing silica aerogel under subcritical	516/101	252/62; 423/335;		Yoldas; Bulent E.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
4	<input type="checkbox"/>	US 5795557 A	19980818	9	Process for the preparation of monolithic silica	423/338			Pajonk; Gerard et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
5	<input type="checkbox"/>	US 5746992 A	19980505	8	Silica aerogel produced under subcritical conditions	423/338	423/335		Yoldas; Bulent E. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
6	<input type="checkbox"/>	US 5738801 A	19980414	4	Hydrophobic silica aerogels	252/62	423/338; 502/233;		Ziegler; Bernd et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
7	<input type="checkbox"/>	US 5656195 A	19970812	6	Moldings containing silica aerogel particles and their	252/62	106/38.3; 106/38.35;		Mielke; Manfred et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8	<input type="checkbox"/>	US 5294480 A	19940315	3	Moldings or sheets made from silica aerogels	442/38	423/335; 423/338;		Mielke; Manfred et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
9	<input type="checkbox"/>	US 5122291 A	19920616	6	Thermal insulating material based on pigment containing	252/62	106/475; 252/587;		Wolff; Bernardo et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
10	<input type="checkbox"/>	US 4954327 A	19900904	7	Production of silica aerogels	423/338	106/18.12; 501/12;		Blount; David H.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
11	<input type="checkbox"/>	US 4432956 A	19840221	9	Preparation of monolithic silica aerogels, the	423/338	423/335; 501/12;		Zarzycki; Jerzy W. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
12	<input type="checkbox"/>	US 4402927 A	19830906	6	Silica aerogel	423/335	423/338		von Dardel; Guy et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

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- L1: (3448) silica adj aerogels
 - L2: (3448) L1
 - L3: (2047) isocyanate adj crosslinks
 - L4: (4) 11 and 13
 - L5: (0) ("s000007").PN.
 - L6: (3448) L1
 - L7: (4675) silica same aerogel
 - L8: (8) 13 and 17
 - L9: (395) acylchloride
 - L10: (3) 11 and 19
 - L11: (497140) silica
 - L13: (7660) aerogel
 - L14: (8) 112 and 113
 - L12: (595) L11 and 13
 - L15: (1721) tmos
 - L16: (0) 112 and 115
 - L17: (26363) teos
 - L18: (1) 112 and 117
 - L19: (9410) tetraethoxysilane
 - L20: (4) 112 and 119
 - L21: (4440) tetramethoxysilane
 - L22: (4) 112 and 121
- Failed
- Saved
- S2: (259464) crosslink\$
 - S3: (7660) aerogel
 - S4: (112) S2 same S3
 - S5: (25594) sol adj gel
 - S6: (41) S4 and S5
 - S7: (3448) silica adj aerogels
 - S8: (710) S2 and S7
 - S9: (0) crosslink\$ adj silica adj aerogels
 - S10: (37) S2 same S7
 - S11: (14) ("6432886") or ("6364953") or ("630
 - S13: (497140) silica
 - S14: (1) S12 and S13
 - S12: (2) S11 and S2
 - S15: (605407) ammonia
 - S16: (1) S14 and S15
 - S17: (8) gelation adj initiator
 - S18: (147785) isocyanate
 - S19: (0) S17 same S18
 - S20: (7091) ammonia same (isocyanate or diisoc
 - S21: (581) S20 same S2
 - S22: (681) ammonia near3 (isocyanate or diisoc
 - S23: (21) S22 same S2
 - S24: (176383) isocyanate or diisocyanate
 - S25: (21645) S24 same S2
 - S26: (37) S7 and S25

triethoxysilane, triethoxysilane, tripropoxysilane, tetramethoxysilane, tetraethoxysilane, tetrapropoxysilane, methyltrimethoxysilane, ethyltrimethoxysilane, propyltrimethoxysilane, methyltriethoxysilane, ethyltriethoxysilane, propyltriethoxysilane, dimethyldimethoxysilane, dimethyldiethoxysilane, diethyldiethoxysilane, gamma-chloropropyltriethoxysilane, gamma-mercaptopropyltrimethoxysilane, gamma-mercaptopropyltriethoxysilane, gamma-aminopropyltriethoxysilane, phenyltrimethoxysilane, phenyltriethoxysilane, phenyltripropoxysilane, diphenyldimethoxysilane, and diphenyldiethoxysilane can be exemplified. Of these, tetramethoxysilane, tetraethoxysilane, methyltrimethoxysilane, ethyltrimethoxysilane, methyltriethoxysilane, ethyltriethoxysilane, dimethyldiethoxysilane, phenyltrimethoxysilane, phenyltriethoxysilane, diphenyldimethoxysilane, and diphenyldiethoxysilane are particularly preferred.

[0076] Compound (ii) may be used alone or two or more kinds may be used in combination. Compound (ii) may be condensed by dehydration after partial hydrolysis. If necessary, trialkylmonoalkoxysilane can be added to compound (ii) for adjusting the physical properties of the reaction product. Although compound (ii) is a compound constituting an inorganic phase in the material of the recording layer of the present invention, for increasing the storage stability of the recording layer material in the state of a solution before being coated on the substrate of the lithographic printing plate precursor of the present invention, it is effective to protect the active metallic hydroxyl group of the inorganic polymer obtained by partial hydrolysis polymerization of compound (ii), e.g., a silanol group (Si-OH). The protection of a silanol group can be effected by esterifying the silanol group (Si-OH) with higher alcohols, e.g., tert-butanol and iso-propyl alcohol. Specifically, the protection can be performed by adding the foregoing alcohols to the organic phase. According to the nature of the inorganic phase, the storage stability of compound (ii) can be further increased at this time by dehydrating the inorganic phase, for example, by means of heating the inorganic phase and distilling off the eliminated water. When acids or bases capable of being catalysts of the hydrolysis polymerization, e.g., hydrochloric acid or ammonia, are present in the inorganic phase, it is also generally effective to lower the concentration of them. This is easily performed by neutralizing the inorganic phase with an acid or a base.

[0077] Compound (ii) is preferably used in an amount of from 3 to 95 wt %, more preferably from 10 to 80 wt %, based on the total solid contents of the recording layer of the lithographic printing plate precursor used in the present invention.

[0078] In the next place, polymers which are obtained by photopolymerization or thermal polymerization reaction to be added, as the crosslinked polymer having functional group (a), to the recording layer of the lithographic printing plate precursor used in the present invention, other than those obtained by a sol/gel reaction, are described below.

[0079] There are methods by photo-dimerization and radical photopolymerization in photo-crosslinking. There are methods by epoxy crosslinking, isocyanate crosslinking and methylol crosslinking in thermal-crosslinking. In any of these methods, a crosslinking reactive group is incorporated into a polymer and crosslinking is performed by the reaction of the polymer by itself or with a monomer.

[0080] In photo-dimerization reaction, crosslinking reaction can be caused by

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Pending
Active
L1: (0) crosslinked adj aerogel
L2: (259464) crosslink\$
L3: (7660) aerogel
L4: (112) 12 same 13
L5: (25594) sol adj gel
L6: (41) 14 and 15
L7: (3448) silica adj aerogels
L8: (710) 12 and 17
L9: (0) crosslink\$ adj silica adj aerogels
L10: (37) 12 same 17
L11: (14) ("6432886") or ("6364953") or ("630
L12: (2) 111 and 12
L13: (497140) silica
L14: (1) 112 and 113
Failed

equally divided into two light components by a beam splitter. One light component is reflected by a fixed mirror and the other light component is reflected by a movable mirror. The reflected light components are recombined by the beam splitter and cause interference due to optical path differences. An interference pattern of the reflected light components, which pass through the stock solution, is observed by a detector. The interference pattern is converted to an infrared spectrum as shown in FIG. 4 by Fourier transformation using a computer.

(11) FIG. 4 shows infrared spectra of a mixed stock solution at 5 minutes and 1 hour after a 30-g tetraethoxysilane (Si(C.sub.2 H.sub.5 O).sub.4) in ethanol solution (silane alkoxide solution) and a 1-g ammonia in 100-g water solution (gelation initiator solution) are mixed. The abscissa indicates the wavenumber and the ordinate indicates the absorbance. The solid line indicates the spectrum at 5 minutes and the broken line indicates the spectrum at 1 hour. The alkoxide solution is hydrolyzed by the effect of the gelation initiator solution to form silanetetraol and then the silanetetraol yields Si--O--Si crosslinks by condensation. The absorbance near 1,035 cm.sup.-1 increases from 50% at 5 minutes to 98% at 1 hour, as shown by an arrow in FIG. 4. Herein, the absorbance is a relative value with respect to the absorbance (100%) of a sample, which is sufficiently gelated, at 6 hours after the mixing. As the gelation proceeds, the absorbance near 1,035 cm.sup.-1 due to the Si--O--Si bonds increases. Thus, the absorbance indicates the degree of gelation.

(12) The degree of gelation of a wet-gel film formed on a substrate is determined, for example, by an apparatus shown in FIG. 5. The basic configuration of this apparatus is the same as that of the apparatus shown in FIG. 3. The degree of gelation is determined by the absorbance at 1,035

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U	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C	P	B
1	US 6364953 B1	20020402	28	Method and apparatus for making aerogel film	118/665	118/52; 118/689;		Kawakami; Nobuyuki et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

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U	Document ID	Issue Date	Pages	Title	Current OR	Current XRef	Retrieval C	Inventor	S	C	P
17	US 6759487 B2	20040706		Thermoplastic polyurethane-silicone	525/478	524/588		Gornowicz; Gerald Alphonse et al.			
18	US 6720368 B2	20040413		Matt, thixotropic paint formulation	523/200	523/205; 523/209;		Field; Rex J.			
19	US 6703071 B2	20040309		Absorbent articles	427/195	427/201; 427/365;		Koslow; Evan E.			
20	US 6676876 B2	20040113		Method of molding a low spin golf ball comprising	264/255	264/275; 264/278;		Sullivan; Michael J. et al.			
21	US 6634963 B1	20031021		Golf ball comprising silicone materials	473/376			Sullivan; Michael J. et al.			
22	US 6569035 B2	20030527		Golf ball comprising silicone material	473/373			Binette; Mark L. et al.			
23	US 6565961 B2	20030520		Absorbent articles	428/304.4	428/308.4; 604/380;		Koslow; Evan			
24	US 6537651 B2	20030325		Heat-transfer label assembly	428/202	428/200; 428/348;		Geurtsen; Friedrich H. et al.			
25	US 6427599 B1	20020806		Pyrotechnic compositions and uses therefore	102/336			Posson; Philip L. et al.			
26	US 6300385 B1	20011009	10	Organic porous material	521/82	521/86; 521/88;		Hashida; Takashi et al.			
27	US 5541234 A	19960730		Process for making low density hydrogel materials	521/66	521/141; 521/155;		Unger; Peter D. et al.			